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अवशेषों के निर्धारण के तरीके
(पहला पुनरीक्षण)

Methods for Determination of
Residue on Chlorination of Tungsten
Metal Powder
(First Revision)

ICS 77.160

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Powder Metallurgical Materials and Products Sectional Committee had been approved by the Metallurgical Engineering Division Council.

The standard was originally published in 1978. In view of the experience gained during these years it was felt necessary to revise the standard again. The following modifications have been made in this revision:

- a) The standard had been drafted to homogenize its structure and wording with other physical test standards.
- b) Reference clause is included.

The composition of the committee responsible for the formulation of this standard is listed in Annex A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***METHODS FOR DETERMINATION OF RESIDUE ON
CHLORINATION OF TUNGSTEN METAL POWDER***(First Revision)***1 SCOPE**

This standard prescribes the method for determination of residue on chlorination of tungsten metal powder which is a measure of the purity of the sample.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

<i>IS No.</i>	<i>Title</i>
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 6492 : 2020	Powders for powder metallurgical purposes — Sampling (<i>first revision</i>)

3 PRINCIPLE OF TEST

Tungsten forms a series of volatile chlorides like WCl_2 , WCl_4 , WCl_5 , and WCl_6 . When a stream of chlorine gas is passed over a heated sample of tungsten metal powder, the metal powder alone is reacted upon forming the volatile chlorides which sublimes at the other end. Impurities like sodium, potassium, calcium and silica are left behind which are weighed by difference in mass of the sample boat and the percentage of residue determined.

4 APPARATUS

The general assembly of the apparatus used shall be as shown in Fig. 1. The use of the various parts is given below:

- Gas bubbler (1) — For the inflow of chlorine gas from Kipp's apparatus and to check the flow rate.
- Connecting tubes (2) — A row of three glass tubes, fitted with ground-glass joints and filled with activated charcoal.

c) Gas bubbler (3) — For drying the chlorine gas by passing through concentrated sulphuric acid.

d) Drying tower (4) — For drying the chlorine gas by passing through anhydrous calcium chloride.

e) Bubbler (5) — For passing the dry chlorine gas through sulphur chloride.

f) Furnace (6) — A 500 mm long, 250 mm dia resistance furnace with 50 mm dia inside muffle with mounting arrangement. The uniform temperature zone of the muffle shall be 250 mm in length; the furnace shall be equipped with a suitable temperature measurement and control equipment [a thermocouple (11)].

g) Quartz tube (7) — A 600 mm long, 300 mm dia quartz tube with ground-glass joints shall be used inside the hot zone of the muffle.

h) Sample boat — A quartz boat of size approximately 80 mm long, 10 mm wide and 40 mm height with a glazed surface shall be used for keeping the sample for chlorination.

j) Empty bottle (8) — It is used for trapping the off gases.

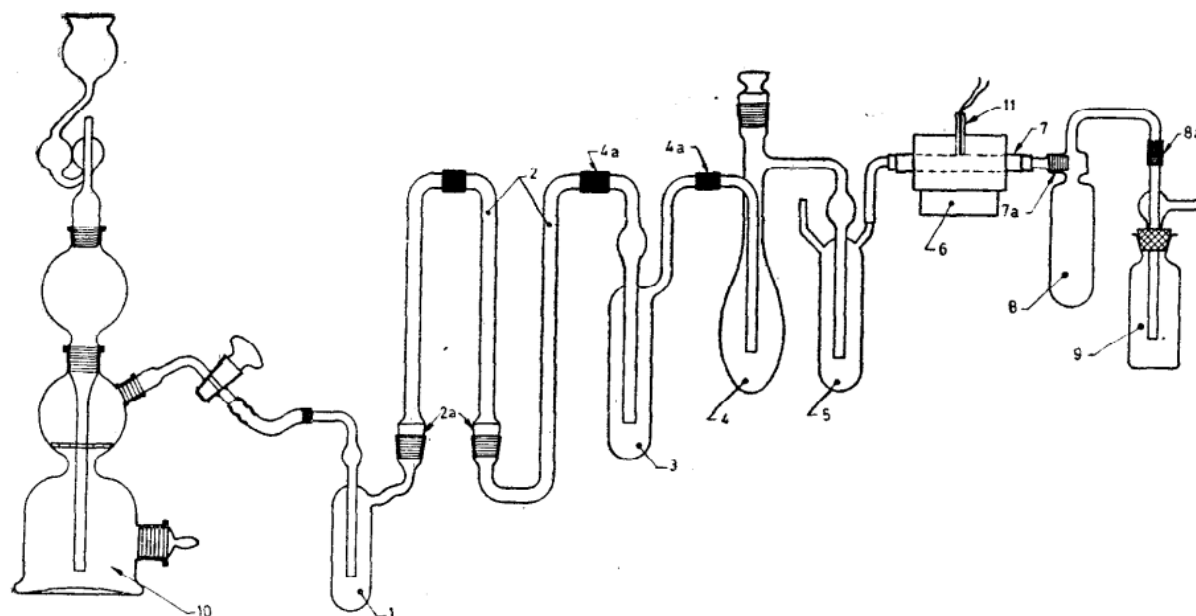
k) Water bottle (9) — The off gases are passed through this bottle and condensed.

m) Kipp's apparatus (10) — For generating chlorine gas in the laboratory at room temperature a medium size Kipp's apparatus shall be used. Alternatively, a chlorine gas cylinder with pressure gauge and regulator may be used.

n) Thermocouple (11) — For recording the furnace temperature.

5 REAGENTS**5.1 Concentrated Sulphuric Acid**

Specific gravity shall be 1.84 (conforming to IS 266). This is used in the bubblers to absorb traces of moisture in the chlorine gas.



Key

- | | |
|--|---|
| 1) Bubbler | 7) Quartz tube |
| 2) Connecting tubes | 7a) Ground glass joint |
| 2a) Ground glass joint | 8) Empty bottle (trap for off gases) |
| 3) Bubbler for H_2SO_4 | 8a) Rubber connector |
| 4) CaCl_2 drying tower | 9) Bottle with water for condensing the off gases |
| 4a) Rubber connector | 10) Kipp's apparatus for generating chlorine gas |
| 5) Bubbler for S_2Cl_2 | 11) Thermocouple |
| 6) Furnace with stand | |

FIG. 1 APPARATUS FOR THE DETERMINATION OF RESIDUE ON CHLORINATION OF TUNGSTEN METAL POWDER

5.2 Activated Charcoal

In granular form of approximately 8 mm size. This is used for filling the three rows of absorption tubes for removal of suspended particles, and hydrochloric acid vapour that are carried with the chlorine gas.

5.3 Sulphur Chloride (S_2Cl_2)

Pure, amber coloured liquid having a specific gravity of 1.70. This is used as carrier for generation of chlorine gas in the cold.

5.4 Potassium Permanganate (KMnO_4)

Solid, small lumps of 6 mm or 12 mm size. This is used for generation of the chlorine gas in the cold.

5.5 Concentrated Hydrochloric Acid

Specific gravity of 1.15 (conforming to IS 265). This is used for generating chlorine gas in the cold.

5.6 Distilled Water

This is used for filling the bottle for collection of the sublimed tungsten chlorides and also as a trap for the off gases.

6 SAMPLING

A representative sample shall be drawn in accordance with IS 6492.

7 PROCEDURE

7.1 The quartz boat shall be thoroughly cleaned and ignited at 700 °C for one hour. It shall be cooled in a desiccator.

7.1.1 The empty boat shall be weighed and 2 g of the sample shall be taken and weighed accurately in a precision balance.

7.1.2 The apparatus shall be assembled. The quartz boat with the sample shall be inserted into the quartz ignition tube and all the connections shall be checked for leak proof joints.

7.1.3 Furnace shall be switched on now. The flow of chlorine gas shall be maintained at a rate of 10 to 15 bubbles per minute until the furnace attains the required temperature. When the temperature reaches 700 °C, the rate of flow of chlorine gas shall be increased for 5 h. After 5 h, the furnace shall be switched off and the rate of flow shall be minimized to 15 bubbles per minute until the furnace cools

down to 100 °C. Now the flow of chlorine gas is stopped. The boat with the residue shall be cooled in a desiccator and weighed. Then the boat shall be thoroughly cleaned to remove the residue and ignited at 700 °C, cooled and weighed. The difference in mass shall give the residue on chlorination for the mass of the sample taken.

8 REPORTING

The percentage of residue on chlorination shall be

calculated by the following formula:

$$\text{Percentage of residue on chlorination} = \frac{A - B}{Y - X} \times 100$$

where

X = mass in g of empty boat,

Y = mass in g of boat with sample,

A = mass in g of boat with residue, and

B = mass in g of boat after cleaning and ignition at 700 °C.

ANNEX A
(Foreword)

COMMITTEE COMPOSITION

Powder Metallurgical materials and Products Sectional Committee, MTD 25

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Indian Institute of Technology Kanpur, Kanpur	DR. ANISH UPADHYAY (<i>Chairperson</i>)
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(METALLURGICAL ENGINEERING), BIS

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